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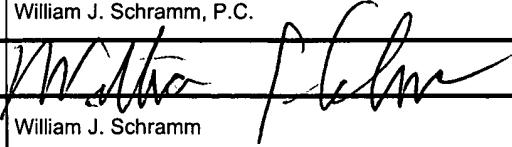
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First Named Inventor	Harjula
Art Unit	1724
Examiner Name	Cintins

Attorney Docket Number 7212.3002.002

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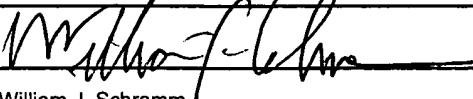
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### SIGNATURE OF APPLICANT, ATTORNEY, OR AGENT

Firm Name	William J. Schramm, P.C.		
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Printed name	William J. Schramm		
Date	December 26, 2006	Reg. No.	24,795

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE  
BOARD OF APPEALS AND INTERFERENCES

APPEAL BRIEF

Applicant: Risto Olavi Harjula

Serial No.: 10/675,138

Filed: September 30, 2003

For: ANTIMONY SILICATE SORBENT  
FOR REMOVAL OF METAL IONS

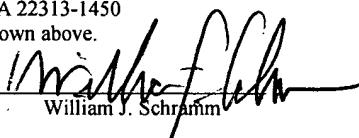
Group Art Unit: 1724

Examiner: Ivars C. Cintins

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**SUPPLEMENTAL APPEAL BRIEF PURSUANT**  
**TO 37 CFR 41.37**

Applicants hereby submit their appeal brief, by and through their attorney, William J. Schramm.

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Section (c) (1) (i): real party in interest

The real party in interest is INEOS SILICAS LTD. of the United Kingdom.

For full disclosure purposes, the research work that is the subject of this application is the result of a research project between British Nuclear Fuels ltd, Crosfield ltd, IVO International, University of Helsinki and the University of Salford. Crosfield changed its name to INEOS SILICAS LTD.

Section (c) (1) (ii): related appeals and interferences

There are no related appeals or interferences.

Section (c) (1) (iii): status of claims

The status of the claims is as follows, with the complete listing of the claims on appeal in the appendix:

Listing of claims:

1. Cancelled
2. Rejected and on appeal
3. Rejected and on appeal
4. Rejected and on appeal
5. Canceled
6. Canceled
7. Rejected and on appeal
8. Canceled
9. Rejected and on appeal
10. Rejected and on appeal
- 11-16. Canceled
- 17-22. Rejected and on appeal

Section (c) (1) (iv): status of amendments

A first amendment after final was not entered as stated in an advisory action mailed June 7, 2006 indicating that the amendment is not in compliance with 37 CFR 1.121

A second amendment after final was not entered as stated in an advisory action mailed July 19, 2006 indicating that the amendment would not be entered due to a claim being dependent upon a canceled claim, *inter alia*.

A third amendment after final was entered as stated in an advisory action mailed August 11, 2006.

Section (c) (1) (v) summary of claimed subject matter

The independent claims are 17 and 22; patentability of claim 9 will also be argued separately.

The examiner has objected that the concise explanation of the subject matter defined in each of the independent claims involved in the appeal has not been referenced in the specification by page and line number. It is respectfully submitted, that the brief does specify where in the specification the independent claims are as to the subject matter. By making reference to specific paragraph numbers, as the appeal brief filed had, there is no other location but specific page and line numbers for paragraph numbers. To simplify the matter, applicant has added the page and line numbers after the paragraph numbers.

As a background for the invention, (paragraph three: page 1, line 14 through page 2, line 3) indicates that in the nuclear industry, large volumes of aqueous streams are produced which contain radionuclides and other polluting metal species. It is desirable to dispose of such wastes with minimum volume to maximize capacity usage.

Antimony silicate has been found to be particularly effective at removing radioactive ions (PP. 15: page 3, line 19 through line 22); also toxic heavy metal ions may be removed. In

particular, antimony silicate doped with one or more elements selected from the group consisting of tungsten, niobium and tantalum has been used. Antimony silicate is used as a sorbent in the removal of metal ions from a liquid medium (PP.30: page, lines 17-20; paragraph 31: page 6, lines 21-25 and paragraph 34, page 7, line 9). See claims 22 and 17.

Another embodiment of the invention is the use of antimony silicate that has been prepared by reacting together in a liquid medium a silicon containing compound, antimony containing compound and a compound containing one or more elements as tungsten, niobium and / or tantalum under polymerization conditions (PP 39: page 8, lines 10-16). Polymerization catalysts include an acid that may be added to the antimony containing compound before the silicon containing compound is added (PP. 27: page, 6, lines 6-10).

Antimony silicate is especially good at removing strontium ions from an acidic aqueous media. Testing has been performed where a Kd value for several nuclides is determined. The value, Kd, is an estimation of the processing capacity of the material. Kd is a measure of the distribution of the isotope between the solid and liquid phases (PP. 18: page 5, lines 8-15).

Several methods of preparation of antimony silicate are described on pages eleven and twelve. Test results for the substantial removal of radionuclides, in particular, strontium, cesium and cobalt are described in figures 9 A. through and including 13.

In the communication of December 4, 2006, the examiner makes reference to section 112 of the patent statute, the sixth paragraph. It is respectfully submitted that the independent claims in this application are not those that fall within section 112, sixth paragraph. The independent claims do not recite “steps plus function” as recited in the patent statute. Further the claims are not general claims but rather are detailed claims and therefore they

do not fall within the sixth paragraph of section 112. *TI Group Automotive Systems (N.A.), Inc. v. VDO North America, LLC*, 375 F.3d 1126, 1135.

Section(c) (1) (vi): grounds of rejection

In the final office action of January 26, 2006 the following rejections were made:

Claimed 22 was rejected under 35 USC 102(e) as being anticipated by U.S. patent 5,858,243 (Bedard). The examiner stated that the '243 patent discloses extracting metal ions (column five, lines 29 – 32) from an aqueous solution (column one, lines 14 – 15) with a silicate material (column two, lines 49 and 53) containing the niobium, tantalum, antimony or mixtures thereof (column two, lines 59 – 61). The examiner further stated that one of ordinary skill in the purification art would, on reading the '243 patent, at once envisage a mixture of antimony and niobium or tantalum as the metal component of the disclosed silicate material.

Claims 2- 4, 17 – 19 and 22 were rejected under 35 USC 103 (a) as being unpatentable over the '243 patent. The examiner stated that the '243 patent discloses removing contaminate metal ions of the type recited (column five, lines 29 – 32) from an aqueous stream (column one, lines 14 – 15) with a silicate material (column two, lines 49 and 53) containing niobium, tantalum, antimony or mixtures thereof (column two, lines of 59 – 61). The examiner further stated that it would have been obvious to one of ordinary skill in the art at the time of the invention was made to select a combination of antimony with niobium or tantalum as constituent "M." in the reference material (column two, lines 59 – 61).

Claims 7, 9, 10, 20 and 21 are rejected under 35 USC 103 (a) as being unpatentable over the '243 reference and further in view of U.S. patent 5,888,398 (Dietz et al.). The examiner stated that the '243 patent discloses the claimed invention with the exception of the recited pH for the aqueous stream, the removal of radioactive metal ions, and the presence of background ions in the stream undergoing treatment. The examiner further

stated that it would have been obvious to one of ordinary skill in the art at the time the invention was made to treat a nuclear waste stream of the type recited ( that is, containing radioactive cesium and background ions such as sodium and calcium) having a pH of less than seven by the process of the'243 patent (column one, line fourteen – 15), since the Dietz reference teaches (column one, lines to 2 – 27) that such nuclear waste streams are typically acidic; and further teaches (column six, lines 3 – 6) that such streams contain background ions such as sodium and calcium.

The examiner further stated, on page four of the final office action, that since applicant contends that the additional materials in the prior art are excluded by the recitation of “consisting essentially of,” applicant has the burden of showing that the introduction of these additional components would materially change the characteristics of applicants’ invention. The examiner argued that absent such a showing, “consisting essentially of” will be construed as equivalent to “comprising”. The examiner argued that since applicant has not provided the above noted showing, the term “consisting essentially of,” recited in claims 17 and 22, has been construed as equivalent to “comprising. “

After a declaration under 37 CFR 1.132 was submitted by applicant by the declaration of Dr. MINIHAN, the examiner found the declaration not persuasive in the advisory opinion of August 11, 2006. The examiner argued that the data presented in the declaration appears to compare tungsten doped antimony silicates against titanium doped antimony silicates for the removal of cesium, strontium and cobalt from nitric acid solutions. The examiner stated that this declaration does not appear to compare antimony silicates doped only with tungsten against antimony silicates doped with both tungsten and titanium; and therefore, this declaration does not show that the presence of titanium in the recited material would materially change the characteristics of applicants’ invention. The examiner stated that accordingly, the “consisting essentially of” language recited in claims 17 and 22 is still being construed as equivalent to “comprising. “

In the concluding paragraph of the advisory action of August 11, 2006, the examiner further stated that since the claims in this application are not limited to tungsten doped

antimony silicates (that is, the antimony silicate could be doped with niobium and/or tantalum), nor are they limited to the removal of cesium, strontium or cobalt from nitric acid solutions, the results presented in the declaration are not commensurate in scope with the claims in the application; and therefore, these results do not show that the presence of titanium in the recited material would materially change the characteristics of applicants' claimed invention, nor do they show new and unexpected results for the claimed subject matter.

**Examiner's comments of December 4, 2006 with respect to noncompliant appeal brief.**

In the communication of December 4, 2006, the examiner indicated that the brief does not contain a concise explanation of the subject matter defined in each of the independent claims involved in the appeal, referring to the specification by page and line number, and to the drawings, if any, by reference characters.

In the communication of December 4, 2006, the examiner indicated that the brief fails to identify for each independent claim involved in the appeal and for each dependent claim argued separately, every means plus function and step plus function under section 112, sixth paragraph and/ or set forth the structure, material, or acts described in the specification as corresponding to each claimed function with reference to the specification as corresponding to each claimed function with reference to the specification by page and line number, and to the drawings, if any, by reference characters.

In the communication of December 4, 2006, the examiner further indicated that a proper heading was needed for each ground of rejection and that only reciting claims 17 and 22 is not deemed to constitute a proper heading; the examiner also objected to the heading containing only claim nine, and is not deemed to be a proper heading for a ground of rejection involving claims 7, 9, 10, 20 and 21.

Applicant will respond to each of the comments made by the examiner in the December 4, 2006 communication in the appropriate section in the brief.

Section (c) (1) (vii): argument

Applicants will argue the patentability of claims 17, 22 and 9.

**I. Claim 22 is not anticipated by BEDARD, United States patent 5,858,243**

**Claim 22 includes the phrase “consisting essentially of”**

The examiner has essentially eliminated the term “consisting essentially of” from the claim in the present case. The examiner relies upon the case, *P. P. G. INDUSTRIES v. GUARDIAN INDUSTRIES*, 156 F. 3d. 1351, 1354 and 1357, 48 USPQ 2d. 1351 (Fed. Cir. 1998) in addition to other pre- Federal circuit or non- Federal Circuit citations on page 3 of the advisory action of August 11, 2006. The PPG case states at page 1354 that by the use of the term “consisting essentially of” the drafter signals that the invention necessarily includes the listed ingredients and is open to unlisted ingredients that do not materially affect the basic and novel properties of the invention. The court continued, in the opinion, supporting the trial court’s claim construction by stating that an ingredient has a material effect on the characteristics of glass “if the effect is of importance or of consequence to those of ordinary skill in the art of glassmaking.” On page 1357 of the opinion, in the example of the PPG case, the glass of Guardian had approximately 40% more material than the preferred embodiment of the patent. The general principle of PPG and its application to the accused composition is applicable here.

For ease of evaluating claim 22, it is reproduced below.

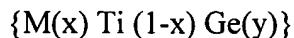
A method of extracting metal ions from an aqueous solution comprising contacting the aqueous solution with a material consisting essentially of antimony silicate doped with one or more elements selected from the group consisting of tungsten, niobium and tantalum.

**The prior art does not disclose antimony silicate doped with tungsten, niobium, and / or tantalum**

Claim 22 indicates that the material consisting essentially of antimony silicate is doped with tungsten, niobium, and/ or tantalum. Such a material is not disclosed in the – 243 patent. That patent could not be described as an antimony silicate because it requires the presence of an alkali metal. See A in the formula in column two. In this case it becomes a molecular sieve material as it contains potassium or sodium salt, inter alia. The material A is always present in the molecular sieve.

In addition, the material disclosed in the'243 patent cannot be described as an antimony silicate as the formula in column two of the'243 patent requires the material to contain an alkali metal, making it into a molecular sieve material in the form of a potassium or sodium salt and not an antimony salt. The dopants of claim 22 are dopants in the silicate of antimony silicate, not replacements for antimony. The material of the'243 patent is a doped sodium or potassium salt, not a doped antimony salt.

Further, in reviewing the '243 patent, column 2, the feature of the disclosure that also pertains to the present application relates to a portion of the formula shown in column 2, lines 26 and 54 and in particular that portion of the formula disclosed below.



When the formula is considered, it is respectfully submitted that titanium must always be present. For example, when y is the smallest amount, 0, then M and titanium are present in equal amounts. When y however is greatest, 0.75, then Ge plus titanium likewise are there.

A review of the working examples of the '243 patent clearly indicates that there is

a requirement that significant amounts of titanium must be present in the compounds utilized and disclosed in the '243 patent. Note in the working examples that a substantial amount of titanium is always used as a portion of the molecular sieve. Note also that the working examples do not contain antimony silicate. Note from examples number 1, 3, 4 & 5 that the majority of the molecular sieve material produced contains sodium, potassium and titanium, none of which are recited in claim 22.

**Test results demonstrate that “consisting essentially of” terminology excludes titanium from antimony silicate doped with tungsten, niobium and/or tantalum.**

Clearly, the disclosure of the'243 patent is not the use of antimony silicate doped with tungsten, niobium and / or tantalum. To further emphasize the distinction between the'243 patent and claim 22, applicants have used the phrase “consisting essentially of” to exclude materials such as titanium. To further support applicants' position of patentability, the declaration of Dr. Alan MINIHAN was submitted. What is important about the declaration is that poor results were obtained utilizing titanium doped antimony silicate. The conclusion reached by the declarant in paragraph seven states:

From these data it was concluded that Ti was an undesirable dopant for antimony silicate to be used for extraction of radioactive metals from acid solution and work on this dopant was not progressed.

The test results supplied clearly demonstrate to one of skill in the art that titanium is undesirable as a dopant for antimony silicate. If it is not satisfactory after testing, why would one spend time and effort to do further testing to prove that it is undesirable? The examiner suggests that normal research personnel should go on and on and on to do some testing without recognizing that such testing is unnecessary, costly and inefficient. The testing that was conducted related to claim 22, and the other claims on appeal. The suggested testing by the examiner is neither necessary nor desirable in today's world of targeted research. The examiner would like to take “an ivory tower” approach to

research. When negative results from research are obtained, further testing is not warranted.

Quite simply, the prior art does not suggest antimony silicate doped with tungsten, niobium and / or tantalum. The test results demonstrate that titanium has a materially detrimental effect on antimony silicate being able to extract metal ions in an aqueous solution. As was stated in the *PPG* case, at 1354, “if the effect is of importance or of consequence to those of ordinary skill in the art of (glassmaking),” then, it is respectfully submitted, that the negative effect should be taken into account in determining patentability. The negative effect of titanium should be taken into account in reaching the conclusion that claim 22 is not anticipated by the’243 patent but rather is patentable in light of the prior art.

**II. Claims 17 and 22 are not obvious over the’243 patent. The examiner indicates that this heading is not satisfactory. The new heading is as follows:**

**Claims 2- 4, 17 – 19 and 22 were rejected under 35 USC 103 (a) as being unpatentable over the’243 patent**

**Applicant is arguing the patentability of independent claims 17 and 22 only.**

For the reasons argued above, claim 22 is not obvious over the’243 patent. The prior art does not disclose antimony silicate doped with tungsten, niobium, and/or tantalum. The use of the terminology “consisting essentially of” excludes titanium. The test results demonstrate that titanium is detrimental to antimony silicate and it is an effect that is of importance or of consequence to one of skill in the art. *PPG* at 1354.

Claim seventeen has all of the features of claim 22. Therefore all the arguments that have been recited above as to claim 22 are equally applicable to claim seventeen. It is respectfully submitted that the examiner has not made a *prima facie* case for obviousness due to the substantial differences between the antimony silicate doped materials of claim

seventeen when compared to molecular sieve of the'243 patent. *In re Fine* 837 F. 2d 1071, 5 USPQ 2d 159 (Fed. Cir. 1988).

Claim seventeen has additional features that are not in the'243 patent. Claim seventeen recites how the antimony silicate doped with tungsten, niobium and/or tantalum is prepared, in particular, in the presence of an acid. Note that the working examples of the'243 patent indicate that the reaction to prepare the molecular sieve is performed in media that has a pH of about 12. Claim seventeen recites the use of an acid. The preparation of materials as called for in claim seventeen is not recited in the'243 patent for it does not suggest the use of an acid for preparing the antimony silicate doped materials

In addition, there is nothing in the'243 patent that would suggest reacting antimony and silicon materials which are further doped with tungsten, niobium and/or tantalum. As indicated above in the argument with respect to claim 22, the major components of the molecular sieve of the'243 patent require the presence of sodium/potassium together with significant amounts of titanium. This molecular sieve is not antimony silicate doped with what is called for in the claims. The art does not suggest or motivate what is called for in the claims. *Gambro Lundia AB v. Baxter Healthcare Corp.* 110 F. 3d 1573, 42 USPQ 2d 1378 (Fed. Cir. 1997). The suggestion must not come from applicants' disclosure. *In re Ruffet* 149 F. 3d 1350, 47 USPQ 2d 1453 (Fed. Cir. 1998). Here the examiner is clearly looking at applicants' claims and disclosure and reaching a conclusion of obviousness in 20-20 "indsight" fashion.

It should be noted that claims 17 and 22 are only rejected over the'243 patent. No secondary reference is used.

### **III.     Claim 9 is not obvious over the'243 patent further in view of Dietz et al (U.S. patent 5,888,398)**

**The examiner has objected to the heading for this section of the brief. The new heading is as follows:**

**Claims 7, 9, 10, 20 and 21 are rejected under 35 USC 103 (a) as being unpatentable over the'243 reference and further in view of U.S. patent 5,888,398 (Dietz et al.).**

**Applicant is arguing the separate patentability of claim 9.**

Claim nine is dependent on claim seventeen and has the further element that the metal ions are radioactive metal ions. All of the arguments presented above with respect to claims 17 and 22 are equally applicable here. If claim seventeen is patentable, then claim nine is patentable.

It is to be noted that the'243 patent does not clearly recite an acidic media (see example two where the solution contains 5.7 M sodium nitrate, 0.6 M sodium hydroxide and a 100mg per liter concentration of cesium chloride solution) nor the treatment of radioactive materials. The examiner attempts to overcome the deficiencies of the'243 patent by relying upon the'398 patent.

The'398 patent discloses a technique for removal of cesium – 137 from nuclear waste streams in an acidic solution. The material that is used is described in column four as a crown ether material, an organic material of highly complex nature. It is believed that this is a classic 20-20 “indsight” combination made by the examiner. The molecular sieve materials of the'243 patent are prepared in alkali media, a pH of about twelve. How does the examiner know that such molecular sieve materials will be stable in the acidic treatment compositions of the'398 reference? It is not stated in the'243 patent. Further, the examiner is substituting an inorganic material of the'243 reference with a highly complicated crown ether organic material of the'398 patent. They are vastly different materials and the'398 patent is working with radioactive materials which are not suggested by the'243 reference. Quite simply, the references are not properly combinable. If the references were combined, they would appear to have a combination of materials that may not be stable at the treatment conditions. Claim 9 surely is patentable!

## **SUMMARY**

It is respectfully submitted that all claims are patentable for the reasons outlined above. It is believed that no fee is due. Authorization is hereby given to charge any additional fees to applicants' attorney's Deposit Account No 50-3865.

Respectfully submitted,

William J. Schramm, P.C.



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Date: December 26, 2006

Section(c) (1) (viii) Claims appendix

1. (Cancelled)
2. A method according to claim 17 wherein the material has a Si: Sb ratio of less than 5.
3. A method according to claim 17 wherein the one or more elements is present in the material at a concentration in the range from about 0.5 to about 30.0 weight %.
4. A method according to claim 2 wherein the one or more elements is present in the material at a concentration in the range from about 0.5 to about 30.0 weight %.
- 5-6. (Cancelled)
7. A method according to claim 17 in which the aqueous solution has a pH <7.
8. (Cancelled)
9. A method according to claim 17 in which the metal ions are radioactive metal ions.
10. A method according to claim 9 in which the radioactive metal ions comprise Sr, Cs, Co, Pu or Am ions.
- 11-16. (Cancelled)
17. A method of extracting metal ions from an aqueous solution comprising contacting the aqueous solution with a material consisting essentially of antimony silicate doped with one or more elements selected from the group consisting of tungsten, niobium and tantalum which material has been obtained by reacting together in a liquid medium a silicon - containing compound, an antimony containing compound and a compound containing one or more of the elements in the presence of an acid.

18. The process of claim 17 wherein the acid is a polymerization catalyst.
19. The method of claim 17 wherein the material is a crystalline antimony silicate material.
20. The method of claim 9 in which the aqueous solution is acidic and contains at least one background ions Na, K, Mg or Ca ions at a higher concentration than the concentration of the radioactive metal ions.
21. The method of claim 9 in which the aqueous solution is acidic and contains at least one background ion Na, K, Mg, or Ca ions, and in which the radioactive metal ions are selectively removed from the aqueous solution, the background ions being left behind in the aqueous solution.
22. A method of extracting metal ions from an aqueous solution comprising contacting the aqueous solution with a material consisting essentially of antimony silicate doped with one or more elements selected from the group consisting of tungsten, niobium and tantalum.

Section (c) (1) (ix) Evidence appendix

The declaration of Dr. Minihan is attached.

Section (c) (1) (x) Appendix

NONE

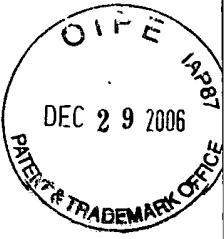
Declaration under 37 CFR 1.132 with regard to US utility patent application number  
10/675,138

I, Alan Reginald Minihan, a British subject of 18 Green Lane, Wallasey, Merseyside, U.K. declare the following:

1. I hold the degree of D. Phil. in Chemistry from University of Oxford and the degree of Master of Arts from University of Oxford. I am a Chartered Chemist and a member of the Royal Society of Chemistry.
2. I am presently employed as Group Product Development Manager by Ineos Silicas Limited, Bank Quay, Warrington, UK and have worked for a total of 21 years on the chemistry and structure of inorganic chemicals for Unilever plc, Crosfield Ltd, and Ineos Silicas Ltd.
3. The research work detailed below was carried out as part of a joint BRITE (EU-sponsored) research project (no F14W-CT95-0016) between British Nuclear Fuels Ltd. (UK), Crosfield Ltd. (UK), IVO International (Finland), University of Helsinki (Finland) University of Salford (UK) between 1<sup>st</sup> January 1996 and 31<sup>st</sup> December 1998. The table of data, Table 26, annexed to this document is from the final report detailing the work carried out in the project.
4. Crosfield Ltd. changed its name to Ineos Silicas Ltd. on 13<sup>th</sup> March 2001.
5. Table 26, which is annexed to this document, shows the distribution coefficients (Kd) for various isotopes in acid solution for tungsten (W)-doped antimony silicates (WSS – samples HMS18) in comparison to antimony silicate (HMS10) and titanium (Ti) doped antimony silicate (HMS19). Description of the preparation of the materials tested is also included in table 26.
6. From the comparative data presented in the table it can be seen that the tungsten doped antimony silicates give much higher values for Kd (e.g 702, 8182, 14251-18303 for HMS18a1; i.e. good extraction behaviour) in comparison to the Kd values obtained for the titanium-doped antimony silicate (22, 187, 1.06). It is believed that the test results indicated herein are representative of the testing program, even though there may be other tests, not included herein, that may have been conducted in the time frame of the program.
7. From these data it was concluded that Ti was an undesirable dopant for antimony silicate to be used for extraction of radioactive metals from acid solution and work on this dopant was not progressed.
8. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



09 May 2006



MP100274 A-US

Signature.....

Date.....09 May 2006.....

Name      Alan Reginald Minihan

Table 26. Distribution coefficients (Kd) for W doped antimony silicates

Sample name	Starting materials	Starting Sb:Si:W ratio	Preparation method	XRD trace	$^{75}\text{Cs}$ Kd [ml/g] in 0.1 M $\text{HNO}_3$	$^{85}\text{Sr}$ Kd [ml/g] in 0.1 M $\text{HNO}_3$	$^{57}\text{Co}$ Kd [ml/g] in 0.1 M $\text{HNO}_3$
1 HMS12 (KSS)	KSB(OH) <sub>6</sub> , TEOS, HNO <sub>3</sub>	1:1 (weighted)	1% mixture at 77°C, overnight	Amorphous	590	19660	1959
2 HMS10 (SbSi)	SbCl <sub>5</sub> in 4 M HCl, Na <sub>2</sub> Si <sub>2</sub> O <sub>5</sub> (Fluka) solutions	1:1 (0.1 M)	1% mixture at 77°C overnight	Crystalline (as antimonite acid)	1354-3702	35515-102700	1509-4282
3 TISS							
4 HMS19 MOSS	HMS10+TiCl <sub>4</sub>	1:1:0.61 (sol)	at 60°C, 1d	Amorphous	22	187	1.06
5 HMS17a1	HMS12 + NH <sub>4</sub> (MoO <sub>4</sub> ) <sub>2</sub>	1:1:1 (weight)	at 77°C, 3 days	Crystalline, AMP?	400	363	285
6 HMS17a2	HMS12 + NH <sub>4</sub> (MoO <sub>4</sub> ) <sub>2</sub>	1:1:0.2 (weight)	—	Amorphous	472	2012	2012
7 HMS17e1	HMS10 + NH <sub>4</sub> (MoO <sub>4</sub> ) <sub>2</sub>	1:1:1:1 (sol)	at 77°C, 2 days	Amorphous	118	141	75
8 HMS17e2	HMS10 + NH <sub>4</sub> (MoO <sub>4</sub> ) <sub>2</sub>	1:2.5:1.7 (sol)	at 77°C, overnight	Amorphous	220	199	116
9 HMS17e3	HMS10 + NH <sub>4</sub> (MoO <sub>4</sub> ) <sub>2</sub>	1:2.5:0.5 (sol)	—	Amorphous	205	118	60
WSS							
3 HMS18al	HMS12 + Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:1:0.5 (weight)	1% mixture at 77°C	Amorphous	702	8182	14251-18303
4 HMS18ald	HMS12 + Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:1:0.3 (weight)	let to dry at 77°C	Amorphous	670	8918	1320
5 HMS18a2	HMS12 + Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:1:1 (weight)	at 77°C overnight	Amorphous	272	2489	251
6 HMS18a3	HMS12 + Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:1:2 (weight)	—	Cryst. Unknown	85.8 (dissolves)	282 (dissolves)	48.5 (dissolves)
7 HMS18a4d	HMS12 + Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:1:0.1 (weight)	let to dry at 77°C	Amorphous	1332	41382	1762
8 HMS18cl	HMS10 + Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:1:1 (weight)	20h at 77°C	Senecyst. SbSi	10632	3252	859

9	HMS18c2	HMS10 +Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:1:1 (weight)	2 days at 77C	Cryst. SbSi	14441	4608	1552
10	HMS18c2d	HMS10 +Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:1:1 (weight)	let to dry at 77C	Cryst. SbSi	17188	35499	1515
11	HMS18c3	HMS10 +Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	0.5:1:1 (weight)	2 days at 77C	Cryst. SbSi	17745	5637	224
12	HMS18c3d	HMS10 +Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	0.5:1:1 (weight)	let to dry at 77C	Cryst. SbSi	20970	8075	640
13	HMS18c4	HMS10 +Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:2.5:1 (sol)	1 d at 77C	Cryst. SbSi	15356	9651	611
14	HMS18c5	HMS10 +Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:2.5:0.5 (sol)	-"	Cryst. SbSi	7008	9492	1101
15	HMS18c6*	HMS10 +Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:1:0.5 (weight)	-"	Cryst. SbSi	18697	42151	1440
16	HMS18c7d	HMS10 +Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:2.5:1.7 (sol)	let to dry at 77C	Cryst. SbSi	6924	5221	289
17	HMS18c8	HMS10 +Na <sub>2</sub> WO <sub>4</sub> *2H <sub>2</sub> O	1:2.5:1.7 (sol)	1 d at 77C	Cryst. SbSi (imp)	3142	435	76.4